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TWIN CITIES MINING RESEARCH CENTER

Walter E. Lewis, Research Director

NASA Contract R-09-040-001

MULTIDISCIPLINARY RESEARCH LEADING TO UTILIZATION OF EXTRATERRESTRIAL RESOURCES

Quarterly Status Report
July 1, 1966 to October 1, 1966

U. S. Bureau of Mines NASA Program of Multidisciplinary Research
Leading to Utilization of Extraterrestrial Resources

QUARTERLY STATUS REPORT July 1, 1966 to October 1, 1966

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STATUS REPORT FIRST QUARTER FISCAL YEAR 1967

U. S. Bureau of Mines NASA Program of Multidisciplinary Research Leading to Utilization of Extraterrestrial Resources

October 1, 1966

Task title: Core group activity
Investigator: Thomas C. Atchison, Senior Research Scientist
Location: Twin Cities Mining Research Center
Minneapolis-St. Paul, Minnesota
Date begun: April 1965 To be completed: Continuing

Personnel: Thomas C. Atchison, Supv. Research Physicist
David E. Fogelson, Research Geophysicist
Clifford W. Schultz, Res. Ext. Metallurgist
Lowell W. Gibbs, Min. Meth. Res. Engineer
Other Bureau personnel, as assigned

PROGRESS REPORT

Objective

To provide the basic scientific and engineering knowledge needed for subsequent development of an extraterrestrial mineral resource extraction, processing and utilization technology for supporting and enhancing the economy of manned lunar and planetary missions.

Progress During the First Quarter

Background information applicable to the program was analyzed and organized, including a considerable backlog of NASA and NASA contractor reports. Particular effort was placed on study of data from Luna 9, Surveyor, and Lunar Orbiter to keep up to date our ideas for rocks that simulate the range of materials most likely to be encountered on the Moon. In general, the new information from these lunar probes has confirmed the appropriateness of our previous selection. Recent discussions with a number of authorities on selenology and volcanology have helped us to complete our selection of simulated lunar materials. Further effort was also placed on literature search for information applicable to some of the experimental tasks for which little information was obtained previously, such as the problem of using explosives on the Moon. A visit to Stanford Research Institute where some work on this problem is being conducted was made near the end of September. Some interesting data on the behavior of explosion products in a vacuum was obtained from Dr. Thomas J. Ahrens and his co-workers in the Shock Wave Physics Division.

During August, an analysis of the purpose and nature of a materials testing laboratory for operation on the lunar surface was made, and an informal report was prepared and submitted to NASA. Also during August, work plans for the current fiscal year for each of the experimental tasks were obtained from the investigator in charge, organized in a uniform style, and presented to NASA as part of the annual status report on the program.

During September, a proposal for continuing the program into its third year was planned. In connection with this, members of the core group visited a number of universities, NASA contractors, and Bureau laboratories in the Western United States. Research groups not previously contacted with whom we discussed our program included those headed by Dr. Harold Masursky, Head of Astrogeologic Studies, U. S. Geological Survey, Menlo Park, California; Dr. Gordon B. Oakeshott, Acting Chief, Division of Mines and Geology, State of California, San Francisco, California; Mr. G. L. Drake, Chief of Life Support Engineering, General Dynamics Corporation, San Diego, California; and Dr. Joseph M. Denney, Director of the Solid State Physics Laboratory, TRW Systems Corporation, Redondo Beach, California.

Status of Manuscripts

"Mining on the Moon", by C. W. Schultz was published in NEW SCIENTIST, July 9, 1966, p. 33.

"Materials Testing Laboratory for Operation on the Lunar Surface", an informal report, by Thomas C. Atchison was submitted to NASA in August.

Task title: Selection and sample collection of simulated lunar materials
Investigator: David E. Fogelson, Project Leader
Location: Twin Cities Mining Research Center
Minneapolis-St. Paul, Minnesota
Date begun: September 1965 To be completed: Continuing

Personnel: David E. Fogelson, Res. Geophysicist
Other Bureau personnel, as assigned

PROGRESS REPORT

Objective

Select and obtain samples of rocks and minerals covering the range of materials likely to be found on the Moon.

Progress During the First Quarter

Five tons of simulated lunar rocks were collected in Oregon and California during September. Our supply of the ten rocks previously selected for the program was replenished, and a granodiorite and two new vesicular basalts were added to our suite of rocks. At the present time we have the following rocks available for use as simulated lunar materials: A granodiorite, a rhyolite, an altered rhyolite, an obsidian, a pumice, a tuff, a dacite, a basalt, a vesicular basalt (with small vesicles), a vesicular basalt (with medium vesicles), a vesicular basalt (with large vesicles), a serpentinite, and a dunite.

The problem of selecting simulated lunar materials was discussed with Dr. Jack Green, Advanced Research Laboratories, Douglas Aircraft Company, Huntington Beach, California; Professor Howel Williams, Geology Department, University of California at Berkeley, Berkeley, California; and Professor Alexander McBirney, Center for Volcanology, University of Oregon, Eugene, Oregon. A number of volcanic rock sites in northern California and Oregon were examined. As a result we plan to replace the previously selected altered rhyolite with an altered latite and to add an andesite and a gabbro to complete our selection of simulated lunar rocks. The main emphasis will remain on volcanic rocks; however, a few coarser grained rocks such as the granodiorite and the gabbro will be of value to other organizations who desire to gain experience with this type of material.

Status of Manuscripts

None in progress.

Task title: Physical properties of simulated lunar materials
Investigator: Thomas C. Atchison, Senior Research Scientist
Location: Twin Cities Mining Research Center
Minneapolis-St. Paul, Minnesota
Date begun: October 1965 To be completed: Continuing

Personnel: All projects are participating

PROGRESS REPORT

Objective

To incorporate simulated lunar materials into basic fragmentation research currently in progress. By this means to determine the composition, elastic, strength, surface, thermal, electrical, magnetic, and explosive shock properties of simulated lunar materials in Earth environment.

Progress During the First Quarter

Measurements of strength and elastic properties of 10 simulated lunar materials were in progress. Pulse velocities in different directions and visible fabric orientations were determined on cubic specimens. Because a number of the rocks exhibit significant anisotropy, spherical specimens were prepared and are being used to identify the axes of symmetry for the materials. When this is done, oriented specimens can be prepared and the effect of the anisotropy on the strength and elastic properties determined.

Pumice cores, 3/4 inch in diameter and 18 inches in length, and disks, 6 inches in diameter and 1 inch in thickness, were prepared and sent to Dr. Werner Goldsmith, Professor of Applied Mechanics, at the University of California at Berkeley. Thomas E. Ricketts, a doctoral candidate supported by the Bureau of Mines and working under Professor Goldsmith, is studying the propagation of stress pulses produced by high velocity impact in rock. Experiments with the pumice show that this porous material exhibits much greater attenuation than the higher density rocks tested previously.

Coefficient of rock strength (Protodyakonov factor) measurements were completed on a number of the simulated materials. This test is a measure of the energy involved in breaking a unit volume of rock to a given size. It is performed by placing a specimen in a tube, dropping a standard weight from a standard height, and measuring the volume of material that is produced below a standard size. Previous measurements of the coefficient of rock strength for a large number of mine rocks have shown correlations with other strength characteristics of the rocks, such as penetration rate for percussive drilling. Measurements thus far completed rank the rocks in order of decreasing strength as follows: Dunite, vesicular basalt, fresh rhyolite, obsidian, and dacite.

Measurements of dielectric constant and dissipation factor in the 20 to 100 MHz range were delayed somewhat because of difficulties with the dielectric sample holder that required returning it to the manufacturer for reconditioning. However, we still expect to complete measurements on 10 simulated lunar rocks during the second quarter.

Status of Manuscripts

None in progress.

Task title:	(1) Chemical reactivity and cold welding of freshly formed surfaces (2) Surface properties of rock in lunar environment
Investigator:	Clifford W. Schultz, Project Leader
Location:	Twin Cities Mining Research Center Minneapolis-St. Paul, Minnesota
Date begun:	January 1966 To be completed: March 1969
Personnel:	Clifford W. Schultz, Res. Extractive Metallurgist William H. Engelmann, Research Chemist Wallace Roepke, Phys. Sci. Technician Sheldon L. Altman, Res. Equipment Operator Ernest Bukofzer, Engineering Technician

PROGRESS REPORT

Objective

Measure the equilibrium constants for the adsorption of gases on the surfaces of silicate minerals. Relate this quantity to the fractional coverage necessary to inhibit cold welding and determine the rate at which various other processes inhibit or prohibit cold welding of vacuum formed surfaces. Extend current experimental studies of surface properties of rocks and minerals to include lunar environment.

Progress During the First Quarter

During the past quarter two significant changes were made in the immediate emphasis of task (1). First, it was decided that a study of the outgassing characteristics of the simulated lunar standards be included in the program. Second, any efforts to study cold welding of fresh surfaces were deferred to a later date. The decision to postpone the cold welding studies was based in part on the need to accommodate the outgassing work and in some measure on the progress made in this area by Dr. Jack Ryan of the Douglas Aircraft Corporation.

The outgassing studies will provide data pertinent to all of the tasks in progress at this Center. This is based on the contention that a valid physical property test can be made only when the sample is equilibrated with its environment. The studies in progress will allow the investigator to estimate the time necessary for a sample of any size or configuration to outgas thoroughly and reach a condition of equilibrium with its surroundings. These studies will also allow us to avoid potentially serious problems with the ion-pumped vacuum systems.

Even more important than these considerations is the fact that the outgassing data, together with data on the equilibrium constants for the adsorption of various gases on rock surfaces, will allow us to estimate the present state of the lunar surface with respect to adsorbed gases.

During the past quarter several observations have been made on a one-inch cube of semiwelded tuff in the NRC oil-pumped system. Some preliminary data are presented in figures 1 and 2. In figure 1 the pump-down curve for the empty chamber is compared with curves for the chamber with the tuff sample in place during the first pump-down and after repeated pumping and backfilling. It can be seen that at first the sample is outgassing and thereby slowing the rate of pressure drop, while after several pump-downs the sample shows an increase in the rate of pressure drop as a result of the lower gas load. In figure 2, in which the pressure rise of the empty closed system is compared with the pressure rise of the system plus sample, it can be seen that the sample is actually acting as a pump which retards pressure rise. The significance of these and similar observations with other samples will not become apparent until a more complete analysis of the data has been made.

The equilibrium constant measurements have been delayed due to failure of the gas analyzer. This equipment has been replaced and is now in operation. In the interim the grinding circuit has been analyzed to determine the optimum operating conditions.

Buildup of equipment capability for the friction studies has progressed rapidly. The Ultek ultrahigh vacuum system (10^{-12} torr) has been received and installed in this last quarter. The National Research Corp. high vacuum (5×10^{-8} torr) system has also been received and acceptance-checked. Several additional items of equipment have been ordered. The items include: An Ultek Quad 250, a quadrapole mass spectrometer for residual gas detection and analysis, an Ewald bench welder for resistance spot welding instrumentation assemblies, a Precision Scientific Co. vacuum oven for initial degassing of fixtures and samples, and an ultrasonic vapor degreaser for cleaning prior to vacuum immersion or vacuum degassing.

Work has progressed satisfactorily on the design of test fixtures and experiments. Bench checking of the test fixtures should be completed next quarter with some preliminary testing performed on the NRC pump stand, providing the vendors meet delivery dates for component parts.

Status of Manuscripts

None in progress.

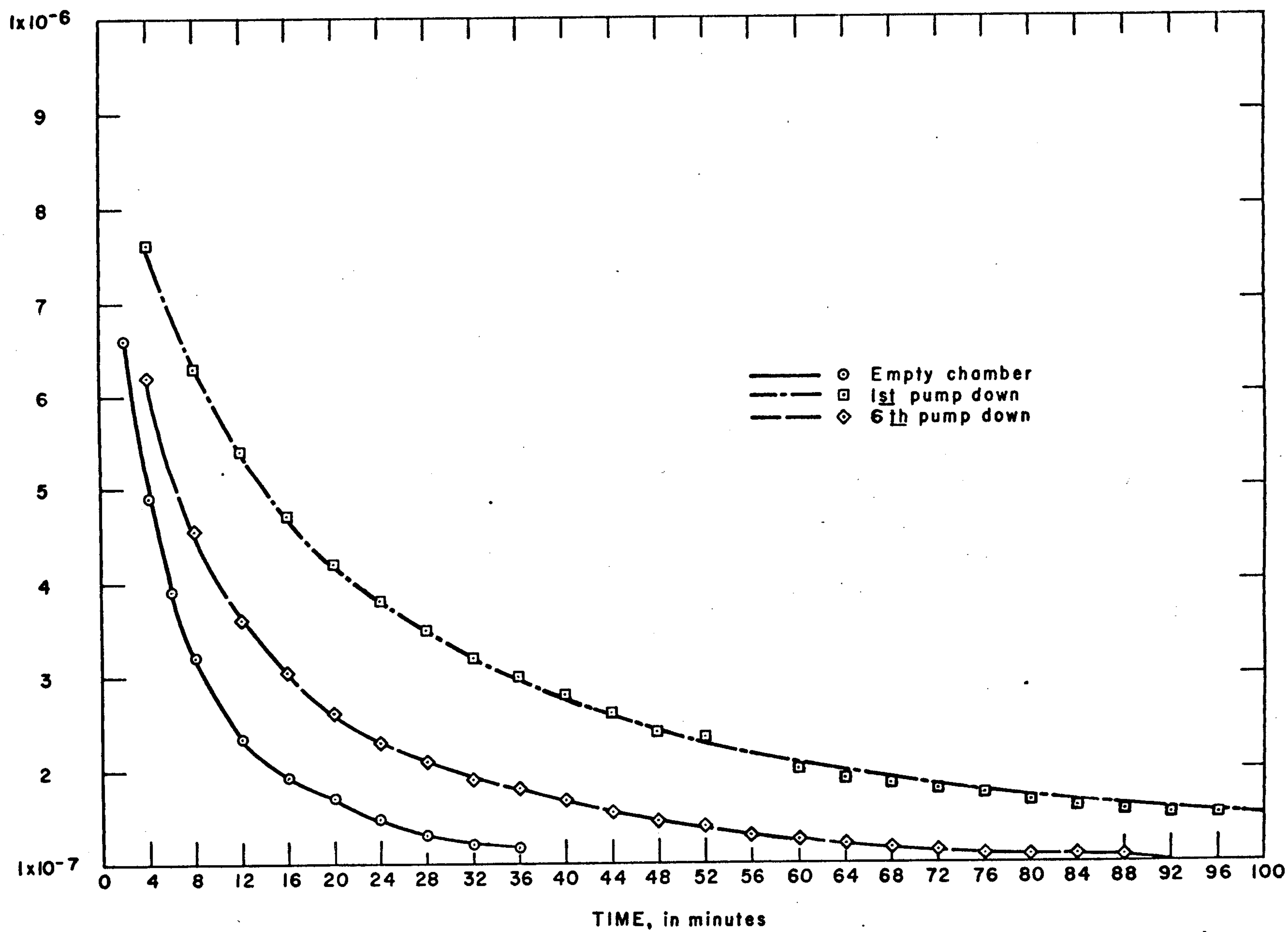

FIGURE 1. - Comparison of pump-down curves for semiwelded tuff and an empty chamber.

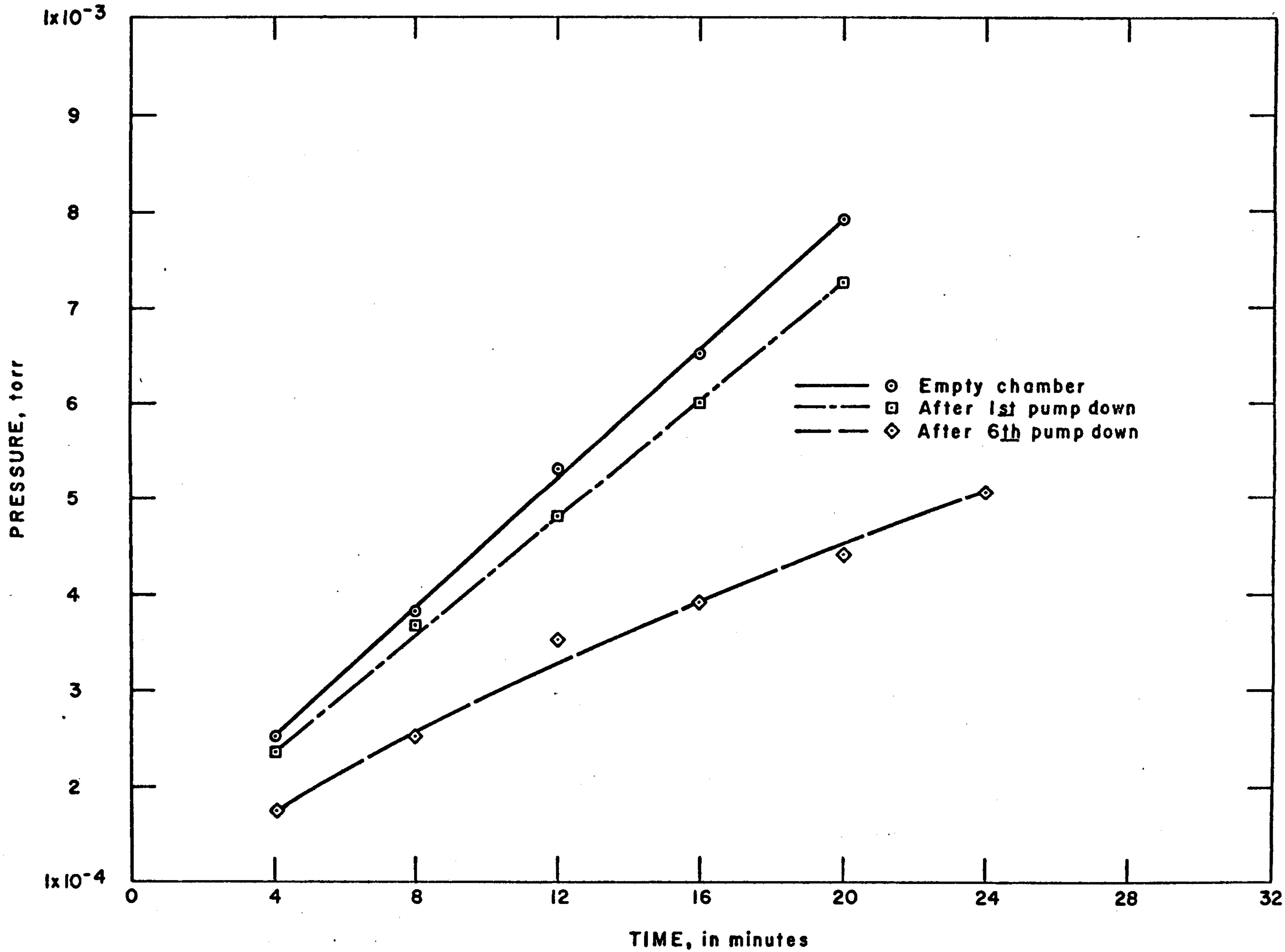

FIGURE 2. - The effect of semiwelded tuff on the rate of pressure rise in a vacuum system.

Task title:	(1) Fracture and other failure mechanisms in lunar environment (2) Strength and elastic properties of rock in lunar environment
Investigator:	John R. McWilliams, Project Leader
Location:	Twin Cities Mining Research Center Minneapolis-St. Paul, Minnesota
Date begun:	June 1966 To be completed: June 1969
Personnel:	John R. McWilliams, Min. Meth. Res. Engineer Robert J. Willard, Geologist Thomas R. Bur, Research Geophysicist Egons R. Podnieks, Mechanical Engineer Richard E. Thill, Geophysicist Peter G. Chamberlain, Geophysicist Kenneth E. Hjelmstad, Geologist Richard M. Brumley, Electronic Technician

PROGRESS REPORT

Objective

Extend current experimental studies of rock failure by such mechanisms as dislocation, twinning, and crack formation to include lunar environment. Extend current measurements of static and dynamic elastic moduli and compressive and tensile strengths of rock to include lunar environment.

Progress During the First Quarter

The first phase of the work represents a partial carryover and extension into the lunar environment of a Bureau of Mines study of the effect of environment on the static and dynamic properties of several rock types including simulated lunar materials. The purpose is to determine the effects, including interactions, of several temperatures (-195°C, 27°C, and 100°C) and atmospheres (air, dry nitrogen, vacuum, and saturated water vapor) on the physical properties (static and dynamic Young's modulus, compressive and indirect tensile strength) of several rock types (basalt, dacite, limestone, and granite).

Tests will be conducted in three groups, A, B, and C. Group A will be directed principally toward the determination of the compressive strength of the several rock types under the environmental conditions outlined above with secondary attention toward obtaining continuous stress, strain, time and pulse velocity measurements up to the fracture point. Tests of Group B cores will be oriented toward determining static Young's modulus and pulse velocity, and Group C samples will comprise a series of indirect (Brazilian) tensile tests to determine the effect of the aforementioned parameters on tensile strength and to provide a source of samples for an exploratory investigation to determine the effect of these conditions on the petrographic features of the rock and on the manner and mode of failure.

Preliminary preparations are virtually complete and environmental testing will be started early in the next quarter.

The Ultek ultrahigh vacuum equipment for use with our loading system has been received and is undergoing acceptance tests. Planning and design work for fixtures, test jigs, and experimental procedures to be used with the chamber are underway.

Status of Manuscripts

None in progress.

Task title:	(1) Rock vaporization, melting, and thermal fracturing methods in vacuum (2) Thermophysical, strength, and elastic properties of rock at elevated and reduced temperatures in vacuum
Investigator:	Robert L. Marovelli, Project Leader
Location:	Twin Cities Mining Research Center Minneapolis-St. Paul, Minnesota
To begin:	October 1966 To be completed: September 1968

Personnel: Robert L. Marovelli, Min. Meth. Res. Engineer
Russell E. Griffin, Electronic Res. Engineer
Ta-Shen Chen, Mechanical Res. Engineer
Carl F. Wingquist, Physicist
David P. Lindroth, Physicist
Sam G. Demou, Physicist
Daryl J. Jersak, Electronic Technician
Walter G. Krawza, Electronic Technician

PROGRESS REPORT

Objective

Investigate the feasibility of extending current thermal fragmentation studies to lunar vacuum environment. Currently the thermophysical, strength, and elastic properties of rock at temperatures up to the melting point are being measured. Extend this work to the low temperature range of lunar environment. Investigate the feasibility of extending these property measurements to lunar vacuum environment.

Progress During the First Quarter

Work on these tasks is scheduled to begin with the second quarter. Work plans for the rest of the fiscal year follow.

SCHEDULE OF WORK

Second Quarter

Start installation and checkout of the thermal shock furnace and vacuum system. Introduce some of the simulated lunar rocks into the conventional work flow in order to examine the temperature sensitivity of selected thermophysical and mechanical properties at atmospheric pressure. Consider modification of the measurement techniques to vacuum environment.

Third Quarter

Complete checkout of the shock furnace and vacuum system. Compare melting temperatures of simulated lunar rocks between atmospheric pressure and 10^{-5} torr during the checkout process. Compare qualitatively the thermal shock fragmentation of these rocks over the temperature-pressure range of the equipment.

Complete preliminary bending strength tests of granite and basalt at atmospheric pressure between room temperature and liquid nitrogen temperature. Design modified apparatus for vacuum tests and prepare test specimens.

Complete measurements of the frequency dependency of the dielectric constants and dissipation factors of the simulated lunar rocks that can be handled with available equipment. Design modified equipment for similar measurements over the lunar temperature range and in vacuum environment.

Subject all the simulated lunar rocks to heat from a plasma torch, rocket type burner, and high-frequency dielectric heater in addition to tests in the thermal shock furnace. Decide which, if any, of the above heat sources would provide meaningful thermal fragmentation data under closely simulated lunar environment.

Fourth Quarter

Continue work of previous quarters with emphasis on the adaptation of present laboratory measurement techniques for the temperature dependence of physical properties to measurements in lunar environment, and on the conduct of rock disintegration tests in lunar environment. If present heat sources prove unsuitable upon completion of third quarter work, consider alternate heat sources that can be operated in connection with vacuum chamber.

MAJOR EQUIPMENT REQUIREMENTS

Thermal shock vacuum furnace (available), high-speed rotary prism camera (available), loading apparatus and Dewar block for low temperature studies (available).

MANUSCRIPT PREPARATION SCHEDULE

Studies of rock strength between room and liquid nitrogen temperatures will probably result in a technical journal article during the third quarter.

Task title:	(1) Cuttings removal in drilling in lunar environment (2) Cooling and lubricating bits in drilling in lunar environment
Investigator:	James Paone, Project Leader
Location:	Twin Cities Mining Research Center Minneapolis-St. Paul, Minnesota
To begin:	January 1967 To be completed: December 1969
Personnel:	James Paone, Min. Meth. Res. Engineer Dick L. Madson, Min. Meth. Res. Engineer Robert L. Schmidt, Mining Engineer Vacancy, Physicist Kenneth G. Pung, Electronic Technician David A. Larson, Engineering Technician

PROGRESS REPORT

Objective

Investigate various means of removing drill cuttings with and without flushing media in lunar environment. Investigate problems of heat removal and bit lubrication associated with drilling in lunar environment.

Progress During the First Quarter

Work on these tasks is scheduled to begin with the third quarter of the fiscal year.

Task title:	Effect of lunar environment on behavior of fine particles
Investigator:	David E. Nicholson, Project Leader
Location:	Spokane Mining Research Laboratory Spokane, Washington
Date begun:	April 1966 To be completed: March 1969
Personnel:	David E. Nicholson, Min. Meth. Res. Engineer William R. Wayment, Min. Meth. Res. Engineer Dennis J. Kelsh, Physical Chemist Robert G. Parker, Chemist Colen S. Smith, Mining Engineer

PROGRESS REPORT

Objective

Extend current studies of fine particle behavior in mine backfill applications to include lunar environment. Measure such properties as density of packing, repose or friction angles, and rates of flow through orifices or channels. This work will be correlated with the study of electrostatic properties of granular particles being conducted at College Park, Maryland.

Progress During the First Quarter

Classification of 300 pounds of commercial ground silica into six selected particle size bands with each band having about 10-micron particle size spread was completed. About fifteen pounds of material were obtained in each particle size band, this sample being sufficient to run a series of tests to determine capillarity, sedimentation volume, permeability, zeta potential, and other properties.

Initial visual examination indicates that the classification is of good quality. There is concern about the contamination of the material processed in the classifier and separated in the cyclone air separator. Carbon and iron are believed to be the two major contaminators. Special coatings, adding a ceramic liner material to the classifier and cyclone, or constructing the classifier of a better metal alloy are being considered before working with the lunar type materials.

Tests and property measurements for each particle size band have been started to determine capillarity, permeability, zeta-potential, shrinkage, and sedimentation volume. The initial test series should be completed in the second quarter.

Background study has been carried out with regard to work by others on lunar soil conditions. Of particular interest are attempts to produce simulated lunar soils in vacuum and inert gas atmosphere.

Additional work performed during the quarter included putting into operation the classifier, zeta meter, and other needed equipment. For the classifier this included installing a compressed air system (auxiliary air supply) with after cooler, air dryer, and filters. A nitrogen storage, pumping, and distribution system (part of a missile fueling system) was obtained with the Atlas Missile Site which we have acquired for use as an auxiliary laboratory. This system is being checked out and evaluated as a possible component of the inert gas mill-classifier system planned for purchase and installation in the fourth quarter.

Status of Manuscripts

None in progress.

Task title:	Support for underground lunar shelter
Investigator:	Ernest L. Corp, Project Leader
Location:	Spokane Mining Research Laboratory Spokane, Washington
Date begun:	April 1966 To be completed: March 1969
Personnel:	Ernest L. Corp, Min. Meth. Res. Engineer Robert C. Bates, Min. Meth. Res. Engineer

PROGRESS REPORT

Objective

The ultimate objective of this project is to advance the ground support technology needed to carry on extraterrestrial mining in support of space missions. The immediate objectives are: (1) To define the problems which will be encountered in designing a lunar ground support or environmental shelter system; (2) to investigate possible materials (indigenous and transported) which can fulfill the requirements for utilization in a support or environmental shelter system; (3) to formulate design concepts for support systems utilizing the most favorable materials.

Progress During the First Quarter

There has been no time devoted to this project during the quarter. Background material pertinent to work on objectives 1 and 2 has been collected but has not been studied. Additional time will be devoted to the project next quarter so that no delay in the overall work plan is anticipated.

Status of Manuscripts

None in progress.

Task title:	(1) Effect of vacuum on explosive properties (2) Effect of micrometeoroid bombardment on explosives (3) Explosive blast effects in lunar environment
Investigator:	Frank C. Gibson, Project Coordinator, Explosive Physics
Location:	Explosives Research Center Pittsburgh, Pennsylvania
Date begun:	July 1966 To be completed: June 1969
Personnel:	Frank C. Gibson, Supv. Research Physicist J. Edmund Hay, Research Physicist Richard W. Watson, Research Physicist William F. Donaldson, Research Physicist Samuel R. Harris, Research Chemist Elva M. Guastini, Explosives Equipment Operator

PROGRESS REPORT

Objective

To develop a body of knowledge relevant to the use of chemical high explosives under lunar environment. Immediate goals are to determine the hazards associated with the storage, handling, and use of explosives in an environment characterized by high vacuum, extreme temperature cycling and a flux of small hypervelocity particles, and to establish techniques for minimizing these hazards.

Progress During the First Quarter

A bombproof facility is being developed for laboratory scale experimentation that is adaptable to both electronic and optical instrumentation, i.e., the bombproof is large enough for Schlieren photography and includes observation ports from the adjacent control room through which events may be viewed by rotating mirror streak photography or single frame photography. A 1.4 mm/μsec streak camera and a Kerr cell camera having modules for 0.05 and 1.0 μsec exposures are available.

It is planned to use several types of vacuum chambers, the selection of which is to be dependent on the immediate requirements of the research, e.g., in determining the effect of the low pressure environment on the behavior of explosives and initiators, small glass vessels are being employed. However, when the rate of growth of, and overpressures generated by, the expanding detonation products are studied, larger blast chambers containing transparent end windows will be utilized. Exploratory experiments on small-scale vessels having mylar ends suggest that this may be a feasible approach since containment of the gases, in large see-through vessels, from detonating charges of sizes that can be adequately instrumented is impractical. A basic design was submitted to the local office of Heraeus-Engelhard Vacuum, Inc., for consideration and evaluation.

A 90-liter/sec Veeco, Inc., oil diffusion pump system with a Baird-Alpert ionization gage and control has been installed for use in the small-scale experiments. A 260-liter/sec Welsch Turbo-Molecular pump system should become available in the second quarter.

Explosives on hand for development and preliminary performance evaluations under lunar conditions include a variety of military types as granular materials and castable solids. Substantial quantities of carefully prepared batches of RDX and HMX, as well as a small amount of a candidate explosive for lunar use are available. The latter, Hexanitrostilbene (HNS), is a development of the Naval Ordnance Laboratory, White Oak, Md., and may be procured from a supplier in North Carolina; charge fabrication techniques for this compound and a similar material, Dipicramide (DIPAM), are currently being explored by NOL. Contact has been established and a visit to the facility during October is planned to derive more information on these materials and their adaptability.

Experimental studies of the effect of low pressure environment on conventional initiators and base charge assemblies are in progress.

Status of Manuscripts

None in progress.

Task title: Volcanism and ore genesis as related to lunar mining
Investigator: Rolland L. Blake, Project Coordinator
Location: Twin Cities Metallurgy Research Center
Minneapolis-St. Paul, Minnesota
Date begun: June 1966 To be completed: May 1967

Personnel: Rolland L. Blake, Research Geologist
Others as assigned

PROGRESS REPORT

Objective

Study the genesis of ore deposits and the occurrence of minerals associated with volcanic activity here on Earth. Study the effects of the lunar environment and other environments on mineralization and ore genesis. Bring together the pertinent information found in the literature on these subjects and define those specific areas where additional work is needed.

Progress During the First Quarter

The literature search was commenced this quarter and a number of reference books on volcanology were found in both the Twin Cities Research Center and the University of Minnesota libraries. Certain books were determined most useful in describing and discussing volcanic processes and the resulting rocks. Several books were ordered for the Bureau library and copies were made of important pages of other books that are out of print.

The investigator attended a seminar on "The Tertiary Volcanic Province of Great Britain," presented at the University of Minnesota by visiting petrology Professor Stuart O. Agrell of the University of Cambridge, England. Subjects covered were a survey of intrusive and extrusive rock relations, and a detailed study of alteration of these rocks by igneous or metamorphic metasomatism (replacement of minerals by simultaneous removal and addition of certain elements). The investigator accompanied Professor Agrell on a two-day field trip through the Cuyuna Iron Range, a portion of which is overlain by volcanic formations.

During the last part of September a field trip was made through northern California and southern Oregon to examine a variety of volcanic sites. At the same time the project was discussed with a number of authorities on volcanology, including Dr. Jack Green, Professor Howel Williams, and Professor Alexander McBirney.

Status of Manuscripts

None in progress.

Task title: (1) Reduction of silicates with carbon
(2) Reduction of silicates in plasma torch
Investigator: Perry L. Weston, Jr., Project Coordinator
Location: Twin Cities Metallurgy Research Center
Minneapolis-St. Paul, Minnesota
Date begun: June 1966 To be completed: May 1969

Personnel: Perry L. Weston, Jr., Supv. Res. Ext. Metallurgist
Larry A. Haas, Research Chemist

PROGRESS REPORT

Objective

The extraction of oxygen from possible lunar raw materials is of prime concern for establishing life survival stations on extraterrestrial bodies. This project is a study of the kinetics and mechanism for reactions between carbon and various oxide silicates in a high temperature vacuum environment. The gaseous oxides of carbon are the prime products in this investigation. Major emphasis of the research will be the establishment of optimum reaction rate criteria. At all stages of the investigation, the relationship between the physical and thermodynamic parameters describing the reaction will be determined. Also, the feasibility of reducing silicates with activated hydrogen in a plasma torch will be studied.

Progress During the First Quarter

According to the literature, the reduction of silica with carbon is not necessarily the simple chemical reaction in equation 1,

$$SiO_{2\,(s)} + 2C_{(s)} \rightleftarrows Si_{(s)} + 2CO_{(g)} \qquad (1)$$

From this equation the rate of reduction could be determined directly by sample-weight loss or measuring the gaseous evolution. However, at 10^{-5} torr and temperatures above 1,000°C, silicon metal apparently will sublime.[1] Partial reduction reactions of silica have been published by Chapman.[2] Chapman observed SiO in the gas phase but reported that it did not exist in the solid phase. Since equation 1 is not the only reaction that can occur, precise analyses of the solid and gaseous phases will be required to determine the reaction kinetics.

[1] Nesmeyanov, An. A. Vapor Pressure of the Elements. Academic Press, N. Y., 1963, p. 461.
[2] Chapman, A. T., G. R. St. Pierre, W. R. Foster, and T. S. Shevlin. Concerning the Stable Phase of Silicon Monoxide. U. S. Dept. Comm., OTS, PB 154,415, 1960, 19 pp.

Assuming that the reaction in equation 1 represents the reactants and products of this experimentation, the equilibrium partial pressure of CO has been calculated at various temperatures according to the published thermodynamic data of Wicks and Block.[3] At 1,247°C (1,520°K), the equilibrium pressure of CO is 1 micron, whereas at 1,349°C (1,622°K) the pressure is 100 microns. Thus, the reaction is thermodynamically feasible in this temperature range provided the environmental pressure is below the equilibrium values. This investigation will be conducted in the temperature range 1,200°C to 1,400°C.

Experimental Apparatus.--The apparatus used in the research has been described in a previous publication.[4] Some modifications of the furnace top assembly, as shown in figure 1, were necessary to obtain optical temperature measurements for the preliminary tests. A new molybdenum resistance heater was also installed in the furnace. Current adjusting type saturable core reactor with a dc null detector and potentiometer are used to power and control the furnace unit. This arrangement is presently being adjusted to control the furnace at constant temperature for long periods of time (24 hours or more). The equipment utilizes a thermocouple feedback balance technique for control. For long periods of operation, a current limiting device was installed in the power circuit to prevent excessive electrical current that could burn out the furnace winding. Vacuum leak rate of the uncharged furnace varies from 0.9 to 9.3 micron liters per minute after bake-out. Future furnace modifications should greatly reduce the leak rate. At 1,375°C, the pressure rise in the system was 7 microns per minute for a 15-gram silica sample and 70 microns per minute for a carbon sample. Considerable difficulty has been experienced in trying to maintain the furnace with sample at low pressure (0.5 micron) and at high temperatures.

Experimental Procedure.--For the tests reported herein, the reactants were minus 400-mesh ultra pure graphite (99.99+ %) and minus 200- plus 270-mesh Fisher reagent grade silica. Dried sample constituents were weighed individually and mixed in a Pitchford Blender for 10 minutes. The sample was then placed in a fused quartz crucible, 35-mm diameter by 75 mm high. After oven drying at 225°F to constant weight, crucible and sample were inserted in the furnace. Before heating, the furnace was evacuated followed by a nitrogen flush and a final evacuation. This procedure reduced the partial pressure of oxygen in the furnace chamber.

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[3]Wicks, C. E., and F. E. Block. Thermodynamic Properties of 65 Elements - Their Oxides, Halides, Carbides, and Nitrides. BuMines Bulletin 605, 1963, 146 pp.

[4]Haas, L. A., and C. W. Schultz. A Torsion Effusion Apparatus for Vapor Pressure Measurement - Vapor Pressure of Silver from 1,200° to 1,500°K. BuMines Rept. of Inv. 6682, 1965, 18 pp.

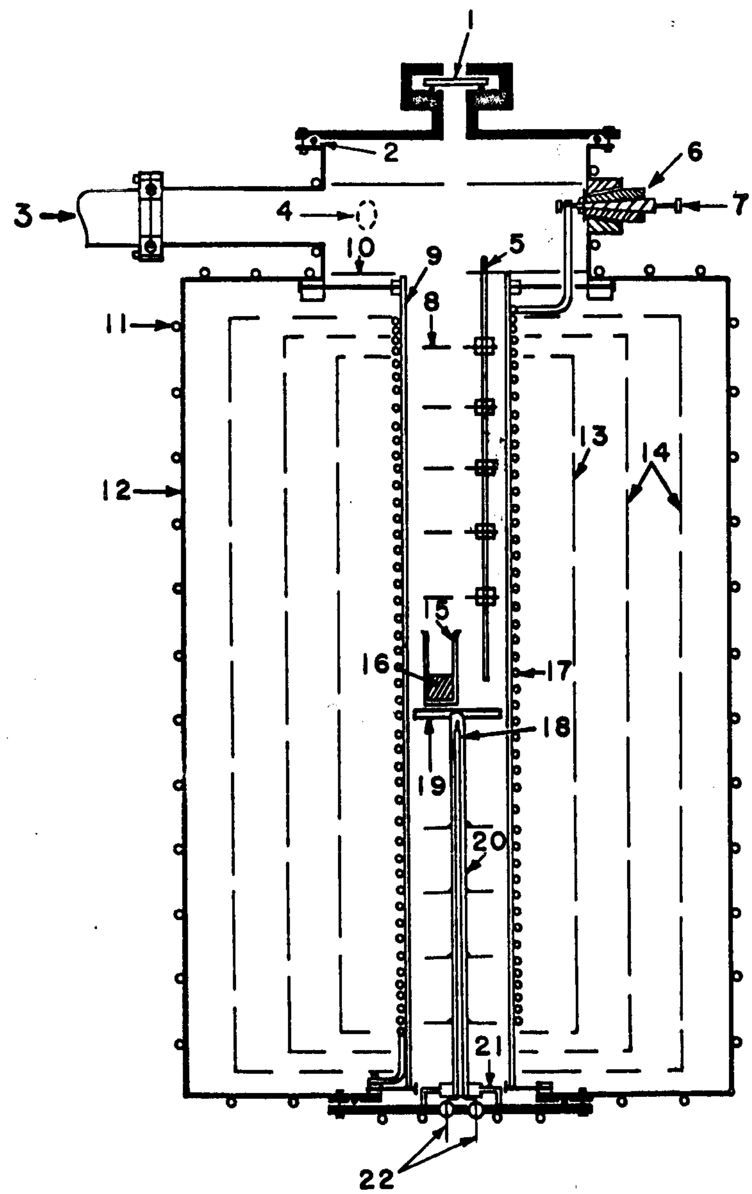

FIGURE 1. - Schematic diagram of the vacuum furnace assembly.

Preliminary tests indicated that some samples may be blown out of the crucible by rapid pump-down of the furnace. A one-quarter inch bleed valve was installed on the forepump line to control the furnace evacuation rate. By keeping the bleeder valve open until the furnace vacuum reaches 1 mm of mercury, the rate of furnace evacuation was essentially halved. With this technique only 7 mg of sample were lost from a 15-gram charge during the nitrogen flush and evacuation cycle. Operating procedure for each test includes 16 hours bake-out at 950°C, a rapid increase to test temperature (usually 2 hours), a 5-hour reaction time and slow cooling overnight to room temperature. The vacuum pumps were in operation throughout the test run.

Experimental Results.--High temperature vacuum experiments have been performed on the individual raw materials. Table 1 shows that the weight loss of silica after 5 hours at 1,400°C was 0.090 gram per 15-gram sample (6 mg loss per gram of silica). The carbon blank showed a little higher weight loss, indicating more adsorbed gas on the sample, or perhaps a small amount of carbon oxidation during the run. The weight loss on a 10-gram sample of carbon at 1,375°C for 5 hours was 0.067 gram (6.7 mg loss per gram of carbon). One-fifth gram moles of carbon and silica were mixed and reacted in the furnace. The results in Test No. 11 indicated that the reaction rate was very slow at temperatures below 1,200°C. At higher temperature, 1,380°C, the reaction rate apparently was so fast that an appreciable sample was blown out of the quartz crucible. Start-up evacuation procedure has been established to minimize sample losses prior to the chemical reaction. High temperature tests are in process to determine the temperature and vacuum at which appreciable sample losses occur.

TABLE 1. - Preliminary test data for the Si-C vacuum reaction

Test No.	Grams of reactants		Temperature °C ± 20	Pressure, microns of Hg	Time, hrs.	Sample, grams	Weight loss, percent
	SiO_2	Carbon					
8	0	10	1375	1.0 to 0.4	5	.067	0.67
9	15	0	1375	0.8 to 0.09	5	.090	0.60
10	12	2.4	975	0.2 to 0.04	5	.017	0.12
11	12	2.4	1170	1.0 to 0.05	5	.092	0.64
14	12	2.4	1225	1.5 to 0.85	5	.174	1.21

Status of Manuscripts

None in progress.

Task title: Magnetic and electrostatic properties of minerals in a vacuum
Investigator: Foster Fraas, Project Leader
Location: College Park Metallurgy Research Center
College Park, Maryland
Date begun: June 1966 To be completed: May 1969

Personnel: Ray A. Heindl, Supv. Chemical Res. Engineer
Foster Fraas, Research Metallurgist

PROGRESS REPORT

Objective

Study adsorption and contact electrification in a vacuum and determine their effect on the separability of nonconducting minerals.

Progress During the First Quarter

Equipment was being designed for contact electrification on vibrating feeders and separation on rotating electrodes. The use of stainless steel as a construction material has been emphasized. Although a pyrex bell jar will initially be used for preliminary observation at pressures down to 10^{-7} torr, the design permits a simple change to a stainless steel bell jar with an ultimate pressure goal of 10^{-9} torr.

Status of Manuscripts

None in progress.

Task title: Biological production of sulfuric acid
Investigator: Joseph A. Sutton, Project Leader
Location: College Park Metallurgy Research Center
College Park, Maryland
Date begun: June 1966 To be completed: May 1967

Personnel: Ray A. Heindl, Supv. Chemical Res. Engineer
Joseph A. Sutton, Research Chemist
John D. Corrick, Research Chemist
Jerry M. Carosella, Microbiologist

PROGRESS REPORT

Objective

Establish the limiting environmental conditions for the survival of bacteria of the genus thiobacillus. Determine the rate of sulfuric acid production within these limits. Conduct a literature survey and visit such laboratories as may be necessary to establish the state of the art in the use of bacteria in any stage of a life support system in an extraterrestrial environment.

Progress During the First Quarter

Tests were conducted to determine the effect pressures ranging from 2.5 to 30 psia have upon the formation of sulfuric acid and cell reproduction. The data showed that pressures below and above 15 psia were detrimental to the formation of sulfuric acid and cell reproduction at a temperature of 25°C. However, further experiments indicated that *T. thiooxidans* could adjust to 30 psia if the organisms were continuously cultivated at this pressure.

Status of Manuscripts

None in progress.

Task title:	Electrowinning of oxygen from silicate rocks
Investigator:	Thomas A. Henrie, Project Coordinator
Location:	Reno Metallurgy Research Center Reno, Nevada
Date begun:	June 1966 To be completed: May 1969
Personnel:	Thomas A. Henrie, Supv. Res. Metallurgist Donald G. Kesterke, Res. Ext. Metallurgist Freddy B. Holloway, Phys. Sci. Technician

PROGRESS REPORT

Objective

To determine the feasibility of obtaining elemental oxygen from silicate minerals by electrolytic methods, for use by the Earth inhabitants of the Moon. Emphasis will be directed toward the determination of essential physical and electrochemical properties of silicate and silicate-base melts containing various amounts of halide salts. Complementary investigations will be made to find suitable nonreactive crucible and anode materials for use in silicate melts, or in melts containing halides.

Progress During the First Quarter

Preliminary efforts were limited to a literature survey and to obtaining various silicate minerals for use in the research program.

Status of Manuscripts

None in progress.

Task title: Stability of hydrous silicates and oxides in lunar environment
Investigator: Hal J. Kelly, Project Coordinator
Location: Albany Metallurgy Research Center
Albany, Oregon
Date begun: April 1966 To be completed: March 1968

Personnel: Hal J. Kelly, Supv. Ceramic Res. Engineer
Raymond L. Carpenter, Research Physicist

PROGRESS REPORT

Objective

The long-range objective is the determination of the energy requirements for dissociating silicate and oxide minerals to recover oxygen and/or water. The immediate objective is to investigate the stability under high vacuum and elevated temperature of some silicate and oxide minerals employing differential thermal analysis (DTA) and thermogravimetric analysis (TGA).

Progress During the First Quarter

The vacuum pumping station has been received and installed. A vacuum of the order of 2×10^{-6} has been obtained without the use of liquid nitrogen trapping. A support to hold the DTA furnace and thermocouples on the base plate of the vacuum pumping station has been designed and is being constructed. When this is completed, the furnace will be installed in the vacuum chamber and calibration runs will be started.

Samples of olivine-series minerals have been obtained for use in the work scheduled for the third quarter. These are being concentrated and the concentrates are being analyzed by X-ray diffraction for structure and iron content.

Review of the literature on DTA of minerals of interest in this project has been started. Several references to DTA studies of minerals in the olivine series have been found. The review has also included references to the use of DTA in determining thermodynamic values.

Status of Manuscripts

None in progress.